

JAPANESE

[JP,2002-025046,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD  
PRIOR ART EFFECT OF THE INVENTION TECHNICAL  
PROBLEM MEANS DESCRIPTION OF DRAWINGS  
DRAWINGS

[Translation done.]

## \* NOTICES \*

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**DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the manufacturing method of the magnetic recording medium which can form a lubricating layer using the lubricant to which the manufacturing method of the magnetic recording medium was started, especially the molecular weight was ready with high precision.

[0002]

[Description of the Prior Art]Generally, the lubricating layer is formed in the top layer of a magnetic recording medium in order to reduce resistance by contact sliding with a recording head.

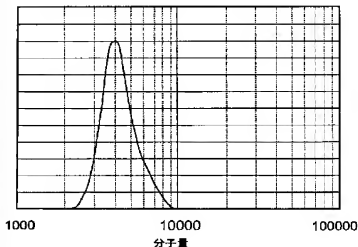
Generally as lubricant to be used, a perfluoropolyether etc. are used.

And the method of forming a lubricating layer with a vacuum deposition method is proposed (JP,7-272268,A). Distribution of the molecular weight spreads [ the commercial perfluoropolyether system lubricant generally used for the vacuum deposition method until now ] from about 1,000 to about 15,000, and a molecular weight degree of dispersion

Drawing selection **Representative draw**

(a)

分子量分布



(b)

Mw	Mn	Mw/Mn
4200	3925	1.07

[Translation done.]

(Mw/Mn) is about about 1.3. Many the low molecular weight constituents and unreacted impurity components whose lubricant of the marketing is a quality unstable ingredient are contained. When heating lubricant like a vacuum deposition method, and making it a vacuum and evaporating lubricant, it evaporates from a low molecule field, evaporates gradually to a polymers field, and is vapor-deposited by the magnetic recording medium. That is, in large molecular weight distribution, therefore a lubricating layer formation process, when a vacuum deposition method is used, an average molecular weight will form a completely different lubricating layer at the production beginning and end. Since it is not producible on the conditions which fixed lubricant cooking temperature, a degree of vacuum, etc. in the low molecule field and the polymers field, control is difficult.

[0003]

[Problem(s) to be Solved by the Invention]For example, in the production on the 1st, in a production initial stage, it is vapor-deposited from the low-molecular-weight field in lubricant, and the lubricant which has the amount field of polymers at the time of the end of production will vapor-deposit to a medium, and a magnetic recording medium with the lubricating layer from which the molecular weight completely differed in the production on the 1st may be produced. In a high temperature atmosphere, since a lubricating layer is low molecular weight, the magnetic recording medium with the lubricating layer of the low-molecular-weight field causes thickness reduction weakly with heat. Therefore, it becomes thin to the set-up thickness, and it gets very bad, and endurance, such as CSS, causes crash, when the worst. Since the lubricating layer of a polymers field has dramatically high viscosity compared with it of a low molecule field, in CSS, the obstacle that a magnetic head will stick to a disk is caused. Therefore, the purpose of this invention is to provide the magnetic recording medium which formed the lubricating layer stable by using for a vacuum deposition method the lubricant to which the molecular weight was ready, and was excellent in endurance under environment with various high-humidity/temperature or low-humidity/temperature etc.

[0004]

[Means for Solving the Problem]The above-mentioned purpose carries out molecular weight refining for lubricant of a commercial perfluoropolyether system to a vacuum deposition method using devices, such as gel permeation chromatography (henceforth GPC), and supercritical extraction (henceforth [ SFE ]), and can attain it by using the refined lubricant for a vacuum deposition method.

[0005]A molecular weight degree of dispersion (Mw/Mn) is made small for lubricant of a commercial perfluoropolyether

system infinite using devices, such as GPC and SFE, and a lubricant film is formed by vapor-depositing it to a magnetic recording medium with vacuum deposition. A molecular weight degree of dispersion shows breadth of a molecular weight of lubricant, and it is expressed by ratio ( $M_w/M_n$ ) of weight average molecular weight ( $M_w$ ) to a number average molecular weight ( $M_n$ ) here. perfluoropolyether system lubricant to be used -- liquid lubricant and a solid lubricant -- whichever may be sufficient and an one end group, a both-ends group, etc. are not limited. What denatured an end group can be used. However, range of weight average molecular weight to 2,000-12,000 is good. When exceeding 12,000 undesirably, and viscosity becomes high, [ since thickness reduction takes place easily into a high temperature atmosphere and CSS durability deteriorates when weight average molecular weight of lubricant is less than 2,000, ] Since a magnetic head will stick to a magnetic disk in CSS, it is not desirable. The range of 4,000-10,000 is more preferably desirable. As for a degree of dispersion ( $M_w/M_n$ ), it is still more desirable that it is 1.1 or less.

[0006]As a means to refine a molecular weight of the above-mentioned perfluoropolyether system lubricant, if GPC, SFE, etc. are used, molecular weight refining can be carried out easily. In addition, a technique will not be asked if molecular weight refining with which it can be satisfied of weight average molecular weight and degrees of dispersion ( $M_w/M_n$ ) which were mentioned above, such as marginal filtration, a distillation-under-reduced-pressure method, thin film distillation, the supercritical chromatography method, and the column chromatography method, can be performed. Especially, since a molecular weight degree of dispersion can obtain 1.1 or less refining lubricant easily, SFE's is preferred. As a subject which vapor-deposits the above-mentioned lubricant, although restriction in particular is not carried out, a magnetic recording medium by which a magnetic layer and a protective layer are usually formed on a substrate or a base film by publicly known methods, such as a hard disk, a floppy (registered trademark) disk, and magnetic tape, is mentioned.

[0007]

[Embodiment of the Invention]Hereafter, an embodiment of the invention is described in detail based on an example.

Drawing 1 is a mimetic diagram of the lubricating layer vacuum evaporator used for this invention, and this device vapor-deposits one side [ every ] lubricant within a vacuum chamber, after vacuum evaporation of one side finishes, it reverses a magnetic disk according to an inversion mechanism, and is vapor-deposited to the remaining fields. A degree of vacuum is controlled by regulation of a flow control variable valve. The heating heater is used as an evaporation

source of lubricant.

[0008]In [drawing 1](#), the petri dish made from stainless steel and 2 1 Lubricant (Fomblin ZDOL2000 [ refined using GPC ]: the weight average molecular weight at this time is 4,000, and Mw/Mn is 1.08), 3 -- as for the career for disks, and 7, a vacuum chamber and 5 are [ the rotary pump for evacuation and 9 ] the turbine pumps for evacuation the variable valve for flow control, and 8 a magnetic disk and 6 the heater for heating, and 4.

[0009](Example 1) As lubricant, the perfluoropolyether system liquid lubricant which performed the above-mentioned molecular weight refining was used. The lubricant 2 is supplied to the petri dish 1 made from 10g stainless steel, and is heated with the heating heater 3. The temperature of the stainless steel petri dish 1 of the source of heating performed temperature control at 80 °C in the sheathed thermocouple. The magnetic disk 5 was installed into the vacuum chamber 4, and the inside of the vacuum chamber 4 was adjusted so that it might be set to  $10^{-5}$ Torr, and it was neglected for 10 seconds. The remaining fields were also made to vapor-deposit lubricant by the retroversion turning point style, it was neglected until it suspended the rotary pump 8 for evacuation, and the turbine pump 9 for evacuation and became atmospheric pressure power after that, and the magnetic disk was removed, and lubricous thickness was measured with a Fourier-transform-infrared-spectroscopy device (FTIR). The lubricous thickness formed on the magnetic disk was 10.0 Å. The sample which formed Cr foundation layer, the CoCrPt magnetic layer, and the carbon protective layer in sputtering one by one on the glass substrate was used for the magnetic disk used in order to form lubricating film.

[0010](Example 2) The weight molecular weight of Fomblin ZDOL2000 refined using the SFE device used 4,200, Mw/Mn used lubricant of 1.07, and it vapor-deposited with the vacuum deposition method.

[0011](Example 3) The weight average molecular weight of Fomblin ZDOL2000 refined using the GPC device used 3,000, Mw/Mn used lubricant of 1.12, and it vapor-deposited with the vacuum deposition method.

[0012](Example 4) The weight average molecular weight of Fomblin ZDOL2000 refined using the GPC device used 4,000, Mw/Mn used lubricant of 1.25, and it vapor-deposited with the vacuum deposition method.

[0013](Comparative example) The sample was produced like Examples 1-4 except vapor-depositing Fomblin ZDOL2000 (2,600 and Mw/Mn are [ weight average molecular weight ] 1.3) which is not refined to the above-mentioned magnetic disk. [Drawing 2](#) indicates the various characteristics (b) to be the molecular weight distribution (a) of lubricant ZDOL2000

of AUSIMONT K.K. of the unrefined lubricant used by the comparative example (Ausimont). 50,000 CSS examinations were done in the environment [ in / using an MR head slider (magnetic resistance type head slider) / for these magnetic disks / 60 \*\*, 80% (high-humidity/temperature), 5 \*\*, and 5% (low-humidity/temperature) ]. A result is shown in Table 1. The load unloading examination of the head in ramped loading and the head which is the durability tests of a magnetic disk was done in 5 \*\* and 5% (low-humidity/temperature) of environment 60 \*\* and 80% (high-humidity/temperature) using the MR head slider. A result is shown in Table 1.

[0014]

[Table 1]

	平均分子量	分散度	CSS	$\mu$	ロード・アンロード試験
実施例 1	4000	1.08	>50K	1.5	>400K
実施例 2	4200	1.07	>50K	1.5	>400K
実施例 3	3000	1.12	>50K	2.0	>400K
実施例 4	4000	1.25	>50K	2.5	>400K
比較例	2500	1.30	20K	4.2	200K

As shown in Table 1, the magnetic disk of an example using the perfluoropolyether refined to vacuum deposition, Compared with the magnetic disk of a comparative example used without refining, the coefficient of friction was small, and it had set to the CSS durability test which is 50,000 times, and the load unloading examination which is 400,000 times, and good endurance was obtained, without adsorbing and crashing.

[0015] Although perfluoropolyether system lubricant was mentioned as the example in the above-mentioned example, any may be sufficient as long as perphloro alkyl polyether, a magnetic recording medium, etc. are already things publicly known as lubricant. The cost can be cut down by the ability of lubricant to be formed in a recording medium as an advantage of a vacuum deposition method without using a solvent. When forming with the dip method, \*\*\*\* of lubricant existed in the disk surface not a little, but the worries do not exist at a vacuum deposition method.

[0016] (Example 5) [Drawing 3](#) indicates the various characteristics (b) to be the molecular weight distribution (a) for every lot of Ausimont ZDOL2000 which refined extracting pressure with supercritical fluid extraction under with 14MPa and a column temperature of 80 \*\* conditions. By refining lubricant shows that molecular weight distribution of narrowing and various characteristics (weight average molecular weight (Mw), a number average molecular weight (Mn), a degree of dispersion (Mw/Mn)) improves so

that drawing 2 and drawing 3 may be compared and understood. moreover -- although the magnetic disk was produced like Example 1 using four sorts of these refined lubricant and the CSS examination and the load unloading examination were done -- adsorption -- good endurance was obtained, without carrying out a head crash.

[0017]

[Effect of the Invention]As mentioned above, according to this invention, since the lubricating layer in which the high precision molecular weight was ready with the vacuum deposition method can be formed on the substrate with which the magnetic layer was formed at least, in various environment, such as high-humidity/temperature and low-humidity/temperature, a magnetic recording medium with good endurance comes to be obtained.

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[Translation done.]